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Investigation of the Kinetics of
Crystallization of Molten Binary
and Ternary Oxide Systems

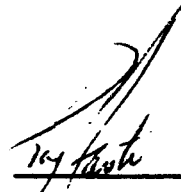
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Investigation of the Kinetics of Crystallization of

Molten Binary and Ternary Oxide Systems

Quarterly Status Report No. 2 - December 1, 1965 through February 28, 1966

Contract No. NASW-1301

SUMMARY

The second quarter's research has concentrated on a broad, although preliminary, examination of all those types of glasses that will be studied in detail in the remaining six months of the initial contractual period. For this purpose, a total of 45 glass compositions have been calculated and prepared with emphasis on systems likely to form complex three-dimensional structures with higher than usual values of elastic moduli. The systems studied include all three systems selected in the proposal, i.e., cordierite glasses with nucleating and anti-nucleating agents, the benitoite system, and the beryl system and, in addition, fluoborate glasses, several of Morey's tantalum oxide-titania (no silica) glasses, and five "invert" glasses. While preparation of these glasses is complete, evaluation of their properties has just begun.

Concurrently with the glass melting research, the additional equipment necessary to carry out an adequate characterization of such glasses is being assembled. A standard model Brookfield viscometer was fitted with elongated tungsten shaft and spindle, calibrated by use of N.B.S. standard viscosity oil "P", and then used to measure the viscosity of three experimental glasses at high temperature. Dynamic measurements of Young's modulus were made on two additional glasses and such measurements were then corroborated by measuring Young's modulus for these glasses using the conventional stress-strain curve, beam deflection, single-point loading technique. Assembly of equipment to permit the dynamic measurement of the shear modulus and Poisson's ratio (using the dynamic Young's modulus values determined in other apparatus) is under way.

INTRODUCTION

This is the second quarterly letter report for Contract NASW-1301, "Investigation of the Kinetics of Crystallization of Molten Binary and Ternary Oxide Systems" covering the period December 1, 1965 through February 28, 1966. The primary objective of this program is to gain a better understanding of glass forming systems by measuring the rate at which crystallization occurs and the effects of anti-nucleating agents on the observed crystallization rates. Determination of the crystallization rate is carried out by continuously measuring the viscosity and electrical conductivity of the molten system as a function of time and temperature with checks of surface tension at selected temperatures. This view of glass formation as a rate phenomenon justifies the consideration of oxide systems previously branded impractical for glass formation and thus allows the search for systems which may yield high strength, high modulus glass fibers to be carried out on an unusually broad basis.

PREPARATION OF GLASS SYSTEMS FOR PRELIMINARY EVALUATION

The first oxide system to be selected for investigation, cordierite or $\text{Mg}_2\text{Al}_4\text{Si}_5\text{O}_{18}$ - a three-dimensional ring silicate structure, has now been melted successfully in 500 gram batches on eight occasions using tungsten or high-purity alumina or beryllia containers. On all occasions a water-white optical grade glass free of seed, stone, and bubbles was obtained. Further, as reported in the first quarterly report under this contract, the glass thus obtained showed an elastic modulus 50% higher than the usual commercial glasses such as fused silica or C.G.W. 7740 as well as yielding experimentally an elastic modulus 10% higher than that calculated using the method of C. J. Phillips (Ref. 1). This glass system seemed ideal for further experimentation and, therefore, the five batches listed in Table I were prepared by adding nucleating and anti-nucleating agents to the system. Evaluation of these glasses is now in progress.

The second oxide system to be studied is benitoite, $\text{BaTiSi}_3\text{O}_9$, whose structure is much like that of cordierite or beryl, comprised of ring ions arranged in sheets with their planes parallel with the metal ions between the sheets and binding together the rings of the different sheets. Although this system has now been melted on several occasions, no glass has as yet been prepared both because of speed of devitrification of the system and because of reaction with the numerous types of crucibles used.

No attempt has yet been made to prepare glasses from oxide mixtures of such proportions that only beryl, a third silicate ring structure, would result. However, three glasses have been melted where beryl should constitute the pre-dominant crystalline phase. Two of these glasses were obtained in optical grade condition but as yet it has proven impossible to cool the third one fast enough to prevent devitrification. Characterization of these glasses through property measurement is under way.

One of the types of glasses which apparently has not been previously studied in connection with glass fiber formation is the fluoborate optical glasses (Refs. 2,3). These glasses contain little or no alkali which according to Phillips' calculations (Ref. 1) contributes to low elastic modulus and very little or no silica. Two such glasses have been prepared and are in the process of evaluation.

A distinctly different type of non-silicate, non-alkali glass system not previously studied by glass fiber research scientists is that built on acid forming elements having relatively high atomic weights. These glasses due to Morey (Ref. 4) and improved for practical manufacture by DePaoli (Ref. 5) may be made, for example, from a mixture of titania and tantalum oxide or from tantalum oxide, zirconia and lanthana. They typically include no alkali and little or no silica and are, therefore, too refractory to be melted in conventional glass-fiber apparatus. Published data (Ref. 6) for similar glasses supports the idea that these glasses have elastic moduli higher than conventional glasses and lack of silica lends hope that such systems may not suffer atmospheric deterioration to the extent common with silicate based glasses. UACRL has melted these glasses on five occasions but has not succeeded in producing glass suitable for evaluation. Further attempts will be made in the third quarter.

An equally novel series of glass systems are those due to Stevels (Ref. 7) and called by him "invert" glasses. These glasses are glasses that do not conform with the Zachariasen three-dimensional network concept which states that at least three corners of each silica tetrahedron is shared so that not more than one corner is shared by a non-bridging oxygen ion, a concept obeyed in general by most commercial silicate glasses used for windows, plate glass, containers, and glass fibers. Stevels introduces a parameter Y_c to express the average number of bridging ions per SiO_4 tetrahedron where

$$Y_c = 6 - \frac{200}{P} \quad \text{and} \quad P = \text{mole \% SiO}_2$$

So that when $P = 33 \frac{1}{3}\%$, $Y = 0$ and the SiO_4 groups are isolated; when $P = 40\%$, $Y = 1$ and on the average SiO_4 groups appear in pairs. These glasses possess the very interesting characteristic that most properties such as viscosity pass through a minimum at $Y = 2$, i.e., 50 mole % SiO_2 . It is possible that the elastic

modulus will likewise pass through such a minimum and will climb steeply as Y decreases from 2 to 1 to 0 but no prior experimental data is available to support this hypothesis. UACRL has accordingly melted the five typical "invert" glasses which are shown in Table II and is currently studying their properties. Of course, many additional "invert" glass systems exist.

PROCEDURES FOR CHARACTERIZING GLASS SYSTEMS INVESTIGATED

The kinetics of crystallization of the glass systems investigated under this contract are determined from continuous measurement of the electrical resistivity and viscosity of the system together with spot checks at temperatures of the surface tension of the molten oxides. These measurements plus the measurement of the elastic modulus of the bulk glass at room temperature serve to characterize the glass system studied. In the first quarterly report the apparatus used to measure the electrical resistivity of the molten oxide system as a function of time and temperature and the devices used to measure Young's modulus of elasticity of the bulk glass by sonic means were described. In this second quarterly report the equipment used to measure the viscosity of the system, the method used to obtain Young's modulus by conventional techniques, and the additional apparatus for measuring the shear modulus are detailed.

VISCOSITY MEASUREMENTS

The device initially used to measure the viscosity of the molten oxide systems at high temperature is the Brookfield Synchro-Electric Viscometer.* The principle of operation of the device is simple. A cylinder or disc or spindle is rotated in the fluid under test through a beryllium-copper spring. The deflection of the spring is read on a dial. The dial reading with the usual disc is multiplied by a simple constant to obtain the resulting viscosity at the particular rotational speed or when special design spindles are used, the device is calibrated through the use of oils of known viscosity. Measurements made at different speeds are used to describe the complete flow properties of the material at hand.

* Trade-mark, Brookfield Engineering Laboratories, Inc., Stoughton, Mass.

The Brookfield viscometer had never been used before at temperatures as high as those likely to occur in this contract. However, this merely meant that the device must be equipped with a long shaft entering the furnace and with a spindle of suitable high temperature material. Tungsten was selected as the material for both the spindle and shaft because of its known compatibility with all the molten oxide systems investigated to date, and Brookfield Engineering Laboratories then designed the tungsten spindle shown in Fig. 1. This tungsten spindle and the Brookfield viscometer were then calibrated using the National Bureau of Standards standard viscosity oil "P" by placing an exact silica replica of the tungsten crucible normally used in the constant temperature bath shown in Fig. 2, filling the silica crucible with oil "P" and running the tungsten spindle in the crucible in such a way as to exactly simulate high temperature operations as shown in Fig. 2. With this constant temperature bath, oil temperatures could be held constant to within ± 0.005 C in the range from -5° to $+107$ C. With this bath, the calibration data obtained for the tungsten spindle is shown in Table III and graphically in Fig. 3.

The viscosity data for N.B.S. standard oil "P" shown as the fourth column of Table III was obtained both by taking the data furnished on the certificate accompanying our shipment of oil "P", plotting it as shown by the solid line of Fig. 4, taking the data furnished in the article published by Shartsis and Spinner (Ref. 8) and plotting it as the dotted line of Fig. 4, and then extrapolating the solid line of Fig. 4 to give a suitably displaced similarly shaped curve. Experience gained in measuring the viscosity of fused silica (Ref. 9) had shown this procedure to be trustworthy. The completed plot of Fig. 4 is then used to furnish the data tabulated in Table IV.

The Brookfield viscometer and tungsten spindle with its elongated shaft were then installed on the tungsten resistance furnace as shown in Fig. 5. The spindle is brought out of the tungsten furnace through a high vacuum fitting. Originally the spindle is at rest, the ground glass previously melted in other furnaces is placed in the crucible, the whole system is then evacuated, flushed with purified argon by loosening the vacuum fitting and allowing the argon (at a positive pressure of 5 in. of water) to stream out, re-evacuated, and then refilled with purified argon. The system is then heated until the glass is molten as judged by visual examination and the tungsten spindle then inserted into the melt and positioned at the proper depth. The temperature of the furnace is adjusted to the desired values and the viscosity of the selected experimental glass is then measured at the various temperatures.

Using the Brookfield viscometer, tungsten spindle, and tungsten furnace the viscosities were measured for three experimental glasses as listed in Table V and plotted in Fig. 6. The experimental glasses evaluated were #1, a cordierite based glass, #30 and #31, soda-silica series glasses with unusually large percentages of zirconia (10.33 and 21.0% by weight, respectively). It will be noted

that all three of these glasses show much steeper temperature-viscosity curves in comparison to a typical commercial boro-silicate glass. Nevertheless, although fiber formation would demand critical temperature control, it should be possible to form fibers successfully using special design recessed apertures.

ELASTIC MODULUS MEASUREMENTS

The sonic equipment described in the first quarterly report was used to measure Young's modulus for the two soda-silica series glasses with the high zirconia contents (10.33 and 21.0% by weight, respectively) and gave the results shown in Table VI. These results thus fail to show the major contribution to increased Young's modulus expected from the high zirconia contents based on the type of calculation made in the paper of C. J. Phillips (Ref. 1). This may be largely due to the detrimental effects of the high soda contents (16.89 and 14.0% by weight, respectively) present in these glasses. An attempt will, therefore, be made to remelt these glasses with decreased alkali additions.

The sonic moduli measurements made of glass #1, cordierite base and similar to batches 4 and 14 reported in the first quarterly report, were checked using measured deflections versus measured loads in three-point loading apparatus equipped with an unusually sensitive load cell. The comparative results of the two methods are shown in Table VII. The values obtained by the sonic method are believed to be more nearly correct since this method gives the slope of the initial part of the stress-strain curve and thus corresponds to a value based on microstresses, while the transverse rupture method essentially yields a value for only very large strains and so corresponds to an average value for a stress-strain curve which is not really a straight line when closely examined. Then too, the sonic method applied to commonly available commercial glasses (as mentioned in the first quarterly report) gave values of Young's modulus in complete agreement with the published values. The values obtained by the transverse-deflection method may also be affected by the fact that the glass samples had been stored two months in laboratory atmosphere before these measurements were made, while the sonic moduli values were measured on freshly ground samples. The importance of such aging can only be evaluated through additional experimentation.

The experimental program at this laboratory stresses the rate concept of glass formation from molten oxide solutions of the proper proportions to yield complex three-dimensional ring structures if the glasses from the melt are allowed to devitrify. It is possible, therefore, that the glasses formed from such melts may show anisotropy. Accordingly, UACRL is setting up equipment for

measuring the shear modulus of the glasses. This determination in conjunction with the sonic determination of Young's modulus will also give values for Poisson's ratio so that any anisotropy in the bulk glass specimen will be apparent.

The equipment now being assembled for the determination of shear modulus is shown in Fig. 7. As indicated, the circuitry measures the velocity of sound in the sample by driving the transmitting piezoelectric crystal with a short burst of radio-frequency vibrations and measuring the transit time of the pulse in the rod. The transmitted pulse is received by a second piezoelectric crystal acoustically coupled to the far end of the sample.

The pulsed oscillator selected emits a gated burst of high-frequency electrical oscillations in the range of 1.4 Mhz to 60 Mhz (1.4×10^6 to 60×10^6 cycles/sec). This voltage drives the transmitting piezoelectric crystal in the compressional mode. This crystal is a wafer 1/8 in. in diameter and 1/64 in. thick acoustically coupled through oil or cement to the glass specimen and so when it vibrates it causes the glass rod to likewise vibrate in the compressional mode.

The receiving crystal is identical to the transmitting crystal and similarly bonded to the rod so that it is mechanically excited by the compressional waves traveling down the glass bar. The time delay between the transmitted and received pulse is then measured on a dual beam oscilloscope and/or an E-put meter. The velocity of this sound wave and the resulting value for the shear modulus of the specimen are then calculated.

CALCULATION OF YOUNG'S MODULUS OF ELASTICITY FROM COMPOSITION OF SIMPLE AND COMPLEX SILICATE GLASSES

In a pioneering paper entitled "Calculation of Young's Modulus of Elasticity from Composition of Simple and Complex Silicate Glasses" (Ref. 1), C. J. Phillips describes a method for calculating Young's modulus of elasticity for some 44 glasses by expressing the content of each oxide in mole percent and multiplying it by a coefficient peculiar to that oxide. Unfortunately, he gives coefficients for only certain oxides likely to be present in glass, namely, SiO_2 , Na_2O , Li_2O , K_2O , B_2O_3 , Al_2O_3 , CaO , MgO , PbO , BaO , ZnO , and BeO . It is hoped that the work of this contract at UACRL can be used to obtain similar values for ZrO_2 , SrO , TiO_2 , Ta_2O_5 , La_2O_3 and other missing oxides. In checking through the paper with our future research in mind, an error was discovered in the coefficient assigned by Phillips to BeO . This error, which has been called to Phillips' attention and with which he agrees, emphasizes the contribution which beryllia is likely to make to high modulus glass

research. We submit two examples to show how this type of calculation is carried out and what the correct value of the coefficient for beryllia should be.

This value for the beryllia coefficient of 19.0 kilobars/mole % replaces the erroneous value of 24.2 kilobars/mole % which results when glass 73 of the Phillips paper is correctly calculated (Phillips through a juxtaposition of the composition had inadvertently obtained the correct value of the beryllia coefficient). This value of the beryllia coefficient would indicate that the attainment of silica-base glasses with a modulus greater than 30×10^6 psi is probably impossible. The calculations, however, do not hold for the non-silicate base glasses such as Morey's glasses and the borofluorate glasses of this report nor can they reasonably be expected to hold for Stevels "invert" glasses.

An extension of the Phillips' method of calculation to Loewenstein's glass Z_1^1 (Ref. 12) shows that zirconia has as high a molal Young's modulus factor as beryllia, a fact not noted prior to this report.

While this value for zirconia is very exciting, initial attempts in this laboratory have failed completely to substantiate it as may be seen from Table V when the experimental values of UACRL glasses 30 and 31 are tabulated. This failure, however, may be solely due to the high alkali content of glasses 30 and 31 in contrast to Loewenstein's glass Z_1^1 which has no alkali present. Further research in this area is thus clearly indicated.

Personnel active on this program during this period were J. F. Bacon, principal investigator and Norman J. Chamberlain, Senior Experimental Technician. They were aided repeatedly throughout the period by Louis J. Tempel, Jr. of the UACRL Instrumentation Section. In addition, Charles E. Shulze of the Materials Sciences Section designed, constructed, and operated the equipment used to obtain Young's modulus by the transverse rupture technique and Herbert G. Aas of the UACRL Instrumentation Section designed the circuitry to be used for shear modulus measurements.

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TABLE I

Preparation of Cordierite Glass with
Nucleating and Anti-Nucleating Additions

<u>Ingredient</u>	Batch No. (amounts of ingredients in grams)					
	<u>1</u>	<u>36</u>	<u>37</u>	<u>38</u>	<u>39</u>	<u>40</u>
Silica	198	149	149	149	149	149
Alumina	120	90	90	90	90	90
Magnesium Carbonate	180	135	135	135	135	135
Beryllium Carbonate	---	135	---	---	---	---
Zirconium Carbonate	---	---	75	---	---	---
Lithium Carbonate	---	---	---	123.5	---	---
Zinc Oxide	---	---	---	---	37.5	---
Cerium Oxalate	---	---	---	---	---	135

TABLE II

Five "Invert" Glass Compositions Recently Prepared

<u>Ingredient</u> <u>(grams)</u>	<u>43</u>	<u>41</u>	Batch No. <u>42</u>	<u>44</u>	<u>45</u>
Silica	176	176	176	176	200
Rutile	16	---	80	48	---
Lead Carbonate	57.6	57.6	57.6	---	---
Potassium Carbonate	75.5	75.5	34.99	75.5	85.75
Calcium Carbonate	78.5	78.5	42.82	78.5	89.20
Strontium Carbonate	62.7	62.7	34.2	62.7	71.25
Barium Carbonate	57.2	57.2	31.2	57.2	65.0
Y _c	1.00	1.45	1.45	1.45	2.0

TABLE III

Calibration of Large Tungsten Spindle in N.B.S. Standard Oil "P"

<u>Viscometer Speed(rpm)</u>	<u>Reading (arb. div.)</u>	<u>Temp. (°C)</u>	<u>Viscosity Oil P (poises)</u>
20.0	74.0	51.0	80
20.0	79.5	47.5	111
20.0	95.7	44.0	145
10.0	37.2	51.0	80
10.0	39.8	47.5	111
10.0	48.1	44.0	145
10.0	73.0	41.0	175
5.0	18.3	51.0	80
5.0	19.6	47.5	111
5.0	24.0	44.0	145
5.0	36.5	41.0	175
5.0	52.0	37.9	214
5.0	60.5	35.0	275
5.0	66.0	32.0	362
5.0	81.1	29.0	450
5.0	82.7	28.0	510
2.5	9.4	51.0	80
2.5	9.75	47.5	111
2.5	12.1	44.1	145
2.5	18.4	41.0	175
2.5	25.5	38.0	214
2.5	30.4	35.0	275
2.5	36.5	32.0	362
2.5	41.4	29.0	450
2.5	41.0	28.0	510
2.5	48.7	27.0	555
2.5	65.2	24.0	780
2.5	81.8	21.0	970
2.5	83.6	18.0	1290

TABLE III (Contd.)

<u>Viscometer Speed(rpm)</u>	<u>Reading (arb. div.)</u>	<u>Temp. (°C)</u>	<u>Viscosity Oil P (poises)</u>
1.0	4.05	51.0	80
1.0	4.45	47.5	111
1.0	4.9	44.0	145
1.0	7.5	41.0	175
1.0	10.1	37.9	214
1.0	12.4	35.0	275
1.0	14.2	32.0	362
1.0	16.4	29.0	450
1.0	16.5	28.0	510
1.0	19.5	27.0	555
1.0	26.1	24.0	780
1.0	32.5	21.0	970
1.0	31.6	18.0	1290
1.0	66.3	14.8	1790
1.0	88.3	12.0	2380
0.5	2.85	44.1	145
0.5	4.2	41.0	175
0.5	5.6	37.9	214
0.5	6.6	35.0	275
0.5	7.7	32.0	362
0.5	8.8	29.0	450
0.5	8.8	28.0	510
0.5	10.0	27.0	555
0.5	13.5	24.0	780
0.5	16.8	21.0	970
0.5	16.8	18.0	1290
0.5	35.5	14.9	1790
0.5	46.75	12.0	2380
0.5	51.2	9.0	off graph used
0.5	57.5	5.85	off graph used

TABLE IV

Extrapolated and Certificate Values of Viscosity for
N.B.S. Viscosity Standard Oil "P"

<u>Temperature °C</u>	<u>Viscosity (poises)</u>	<u>Temperature °C</u>	<u>Viscosity (poises)</u>
30.0	417.8 certif.	33.36	329 (Ref. 8)
40.0	183.3 certif.	29.50	448 (Ref. 8)
50.0	86.6 certif.	26.98	569 (Ref. 8)
		11.98	2,439 (Ref. 8)
		19.10	1,193 (Ref. 8)
		11.81	2,499 (Ref. 8)

TABLE V

Viscosity Measurements for Three Experimental Glasses

Glass Batch #1		Glass Batch #30		Glass Batch #31	
<u>Temp. °C</u>	<u>Viscosity (poises)</u>	<u>Temp. °C</u>	<u>Viscosity (poises)</u>	<u>Temp. °C</u>	<u>Viscosity (poises)</u>
1281	2150	1247	1310	1358	1043
1305	335	1267	954	1378	947
1313	184	1292	765	1405	748
1333	160	1305	557	1420	475
1348	87	1327	365	1445	373
		1342	281	1455	270
		1377	193	1483	213
		1392	186	1492	175
				1508	170
				1532	147
				1558	57

TABLE VI

Sonic Moduli for High Zirconia Glasses

<u>Specimen</u>	<u>Young's Modulus (pounds/in² x 10⁶)</u>	<u>Specimen</u>	<u>Young's Modulus (pounds/in² x 10⁶)</u>
Batch 30 - #1	10.15	Batch 30 - # 9	10.05
Batch 30 - #2	10.18	Batch 30 - #10	10.17
Batch 30 - #3	10.17	Batch 30 - #11	10.38
Batch 30 - #4	10.0	Batch 30 - #12	10.38
Batch 30 - #5	10.33	Batch 31 - # 1	11.55
Batch 30 - #6	10.35	Batch 31 - # 2	11.45
Batch 30 - #7	9.94	Batch 31 - # 3	11.87
Batch 30 - #8	10.23	Batch 31 - # 4	11.25

TABLE VII

Comparative Results of Sonic and Transverse Rupture Determinations of
Young's Modulus for Glass #1

<u>Specimen</u>	<u>Sonic Modulus</u> <u>(pounds/in² x 10⁶)</u>	<u>Transverse Rupture Value</u> <u>(pounds/in² x 10⁶)</u>
1	14.5	13.79
2	14.8	14.50
3	15.1	14.06
4	15.2	14.33
5	14.55	13.92
6	15.38	13.55
7	14.58	13.81
9	15.0	13.76
10	14.4	13.20
11	15.36	14.50
Average	14.89	13.94
Average Deviation	± 0.32	± 0.32

TABLE VIII

Calculations of Young's Modulus for Owens-
Corning Experimental Glass OCX-2124 (Ref. 10)

<u>Constituent</u>	<u>Wt. %</u>	<u>Mole Wt.</u>	<u>Mole Fraction</u>	<u>Mole %</u>	<u>Kilobars/Mole %</u>	<u>Contribution</u>
SiO ₂	71.1	60.06	1.185	70.0	7.3	511
Al ₂ O ₃	21.5	101.94	0.211	12.5	12.1	151
BeO	7.4	25.02	0.296	17.4	X	17.4X
						<hr/> 662 + 17.4X

But Owens-Corning (Ref. 10) achieved 14.4×10^6 psi or 992 kilobars

$$\therefore \text{BeO factor} = \frac{992 - 662}{17.4} = 19.0 \text{ kilobars/mole \%}$$

TABLE IX

Calculation of Young's Modulus for Example 4,
U.S. Patent 3,127,277 (Ref. 11)

<u>Constituent</u>	<u>Wt. %</u>	<u>Mole Wt.</u>	<u>Mole Fraction</u>	<u>Mole %</u>	<u>Kilobars/Mole %</u>	<u>Contribution</u>
SiO ₂	51	60.06	0.850	42.5	7.3	310
CaO	13	56.08	0.232	11.6	12.6	146
MgO	9	40.32	0.223	11.1	12.0	133
BeO	11	25.02	0.440	22.0	X	22X
ZrO ₂	2	123.22	0.016	0.8	28.6*	23
TiO ₂	8	79.90	0.100	5.0	13.3	66.5
Li ₂ O	3	29.88	0.100	5.0	7.0	35
CeO ₂	3	172.13	0.018	0.9	13	12
						725.5 + 22X

*See Table X

But Owens-Corning achieved 16.6×10^6 psi or 1144 kilobars

$$\therefore \text{BeO factor} = \frac{1144 - 725.5}{22} = 19.0 \text{ kilobars/mole \%}$$

TABLE X

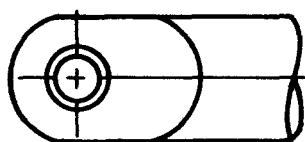
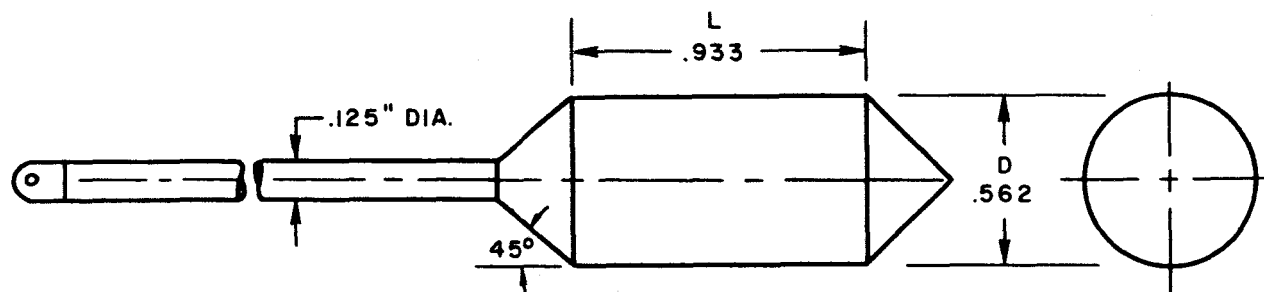
Calculation of Young's Modulus for Loewenstein's Glass Z_1^1

Constituent	Wt. %	Mole Wt.	Mole Fraction	Mole %	Kilobars/Mole %	Contribution
SiO ₂	50.0	60.06	0.833	50	7.3	365
Al ₂ O ₃	5.0	101.94	0.049	2.93	12.1	35.4
ZrO ₂	12.0	123.22	0.097	5.82	Y	5.82Y
MgO	14.0	40.32	0.347	20.82	12.0	250
CaO	19.0	56.08	0.339	20.83	12.6	256
						<hr/> 912.2 + 5.82Y

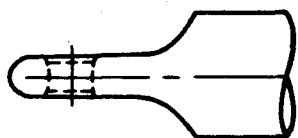
But Z_1^1 (exp. value) = $11 \times 10^5 \text{ kg/cm}^2 = 1078 \text{ kilobars}$

$$\therefore \text{ZrO}_2 = \frac{166}{5.82} = 28.6 \text{ kilobars/mole \%}$$

LARGE TUNGSTEN SPINDLE USED FOR HIGH TEMPERATURE VISCOSITY MEASUREMENT

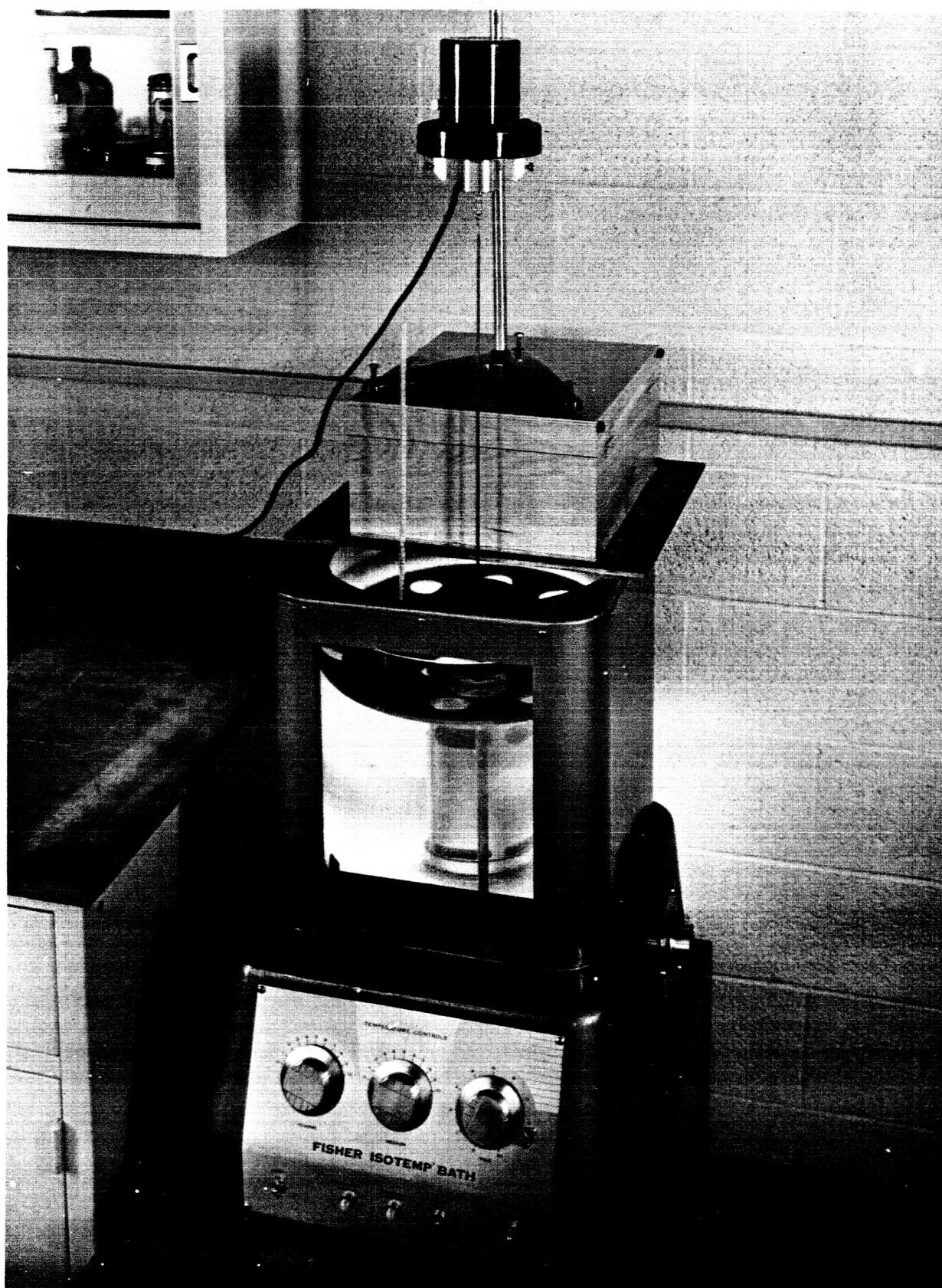


BROOKFIELD VISCOMETER MODEL: RVT
CONTAINER DIAMETER: 2"
MINIMUM CONTAINER DEPTH: 2"



<u>SPEED (R.P.M.)</u>	<u>RANGE (CPS)</u>
100	0 - 3000
50	6000
20	15,000
10	30,000
5	60,000
2.5	120,000
1	150,000
0.5	600,000

BROOKFIELD VISCOMETER AND CONSTANT TEMPERATURE
BATH USED FOR CALIBRATION



CALIBRATION DATA FOR LARGE TUNGSTEN SPINDLE, BROOKFIELD VISCOMETER,
AND STANDARD OIL "P" FROM N.B.S.

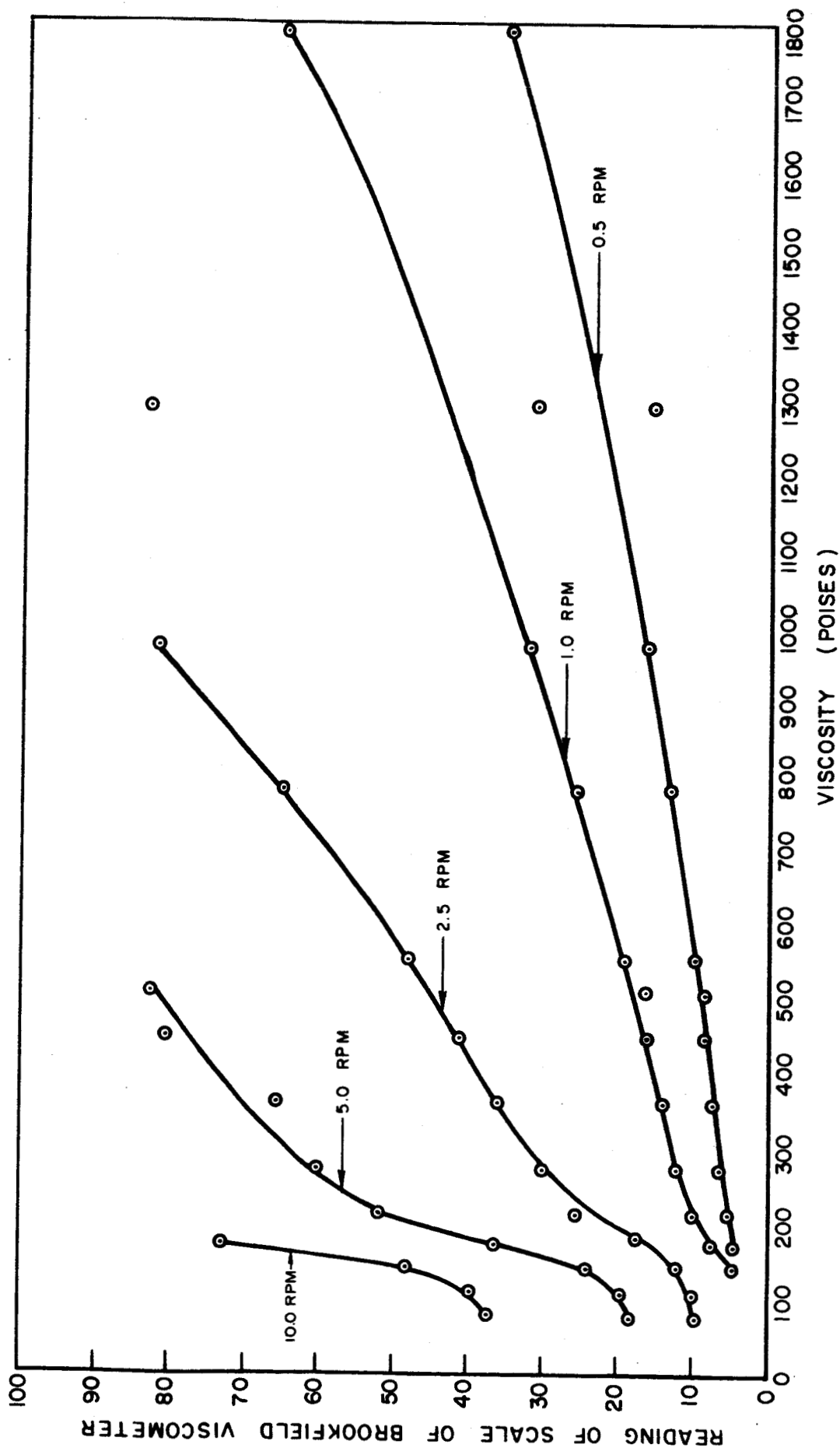
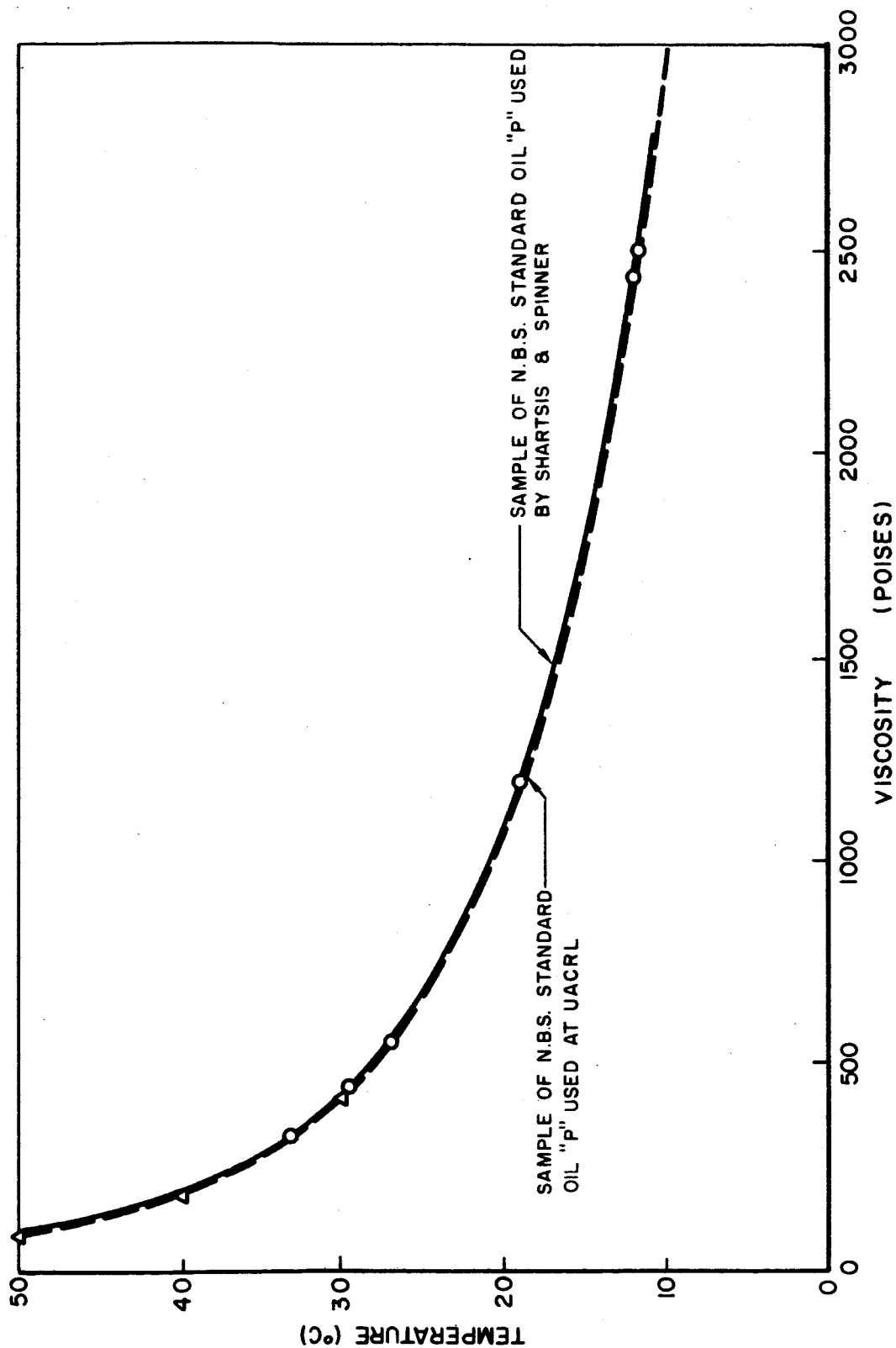
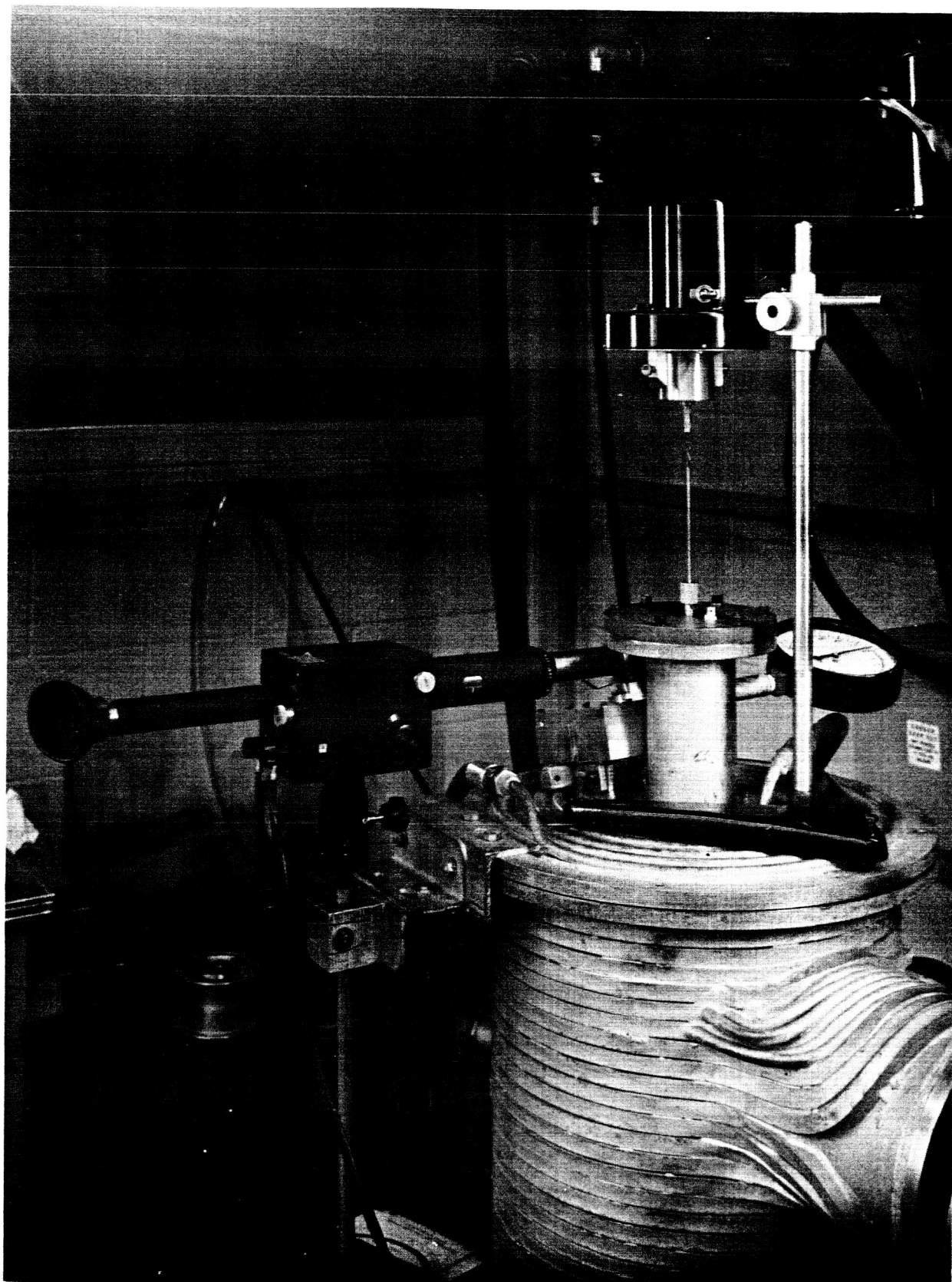


FIG.3

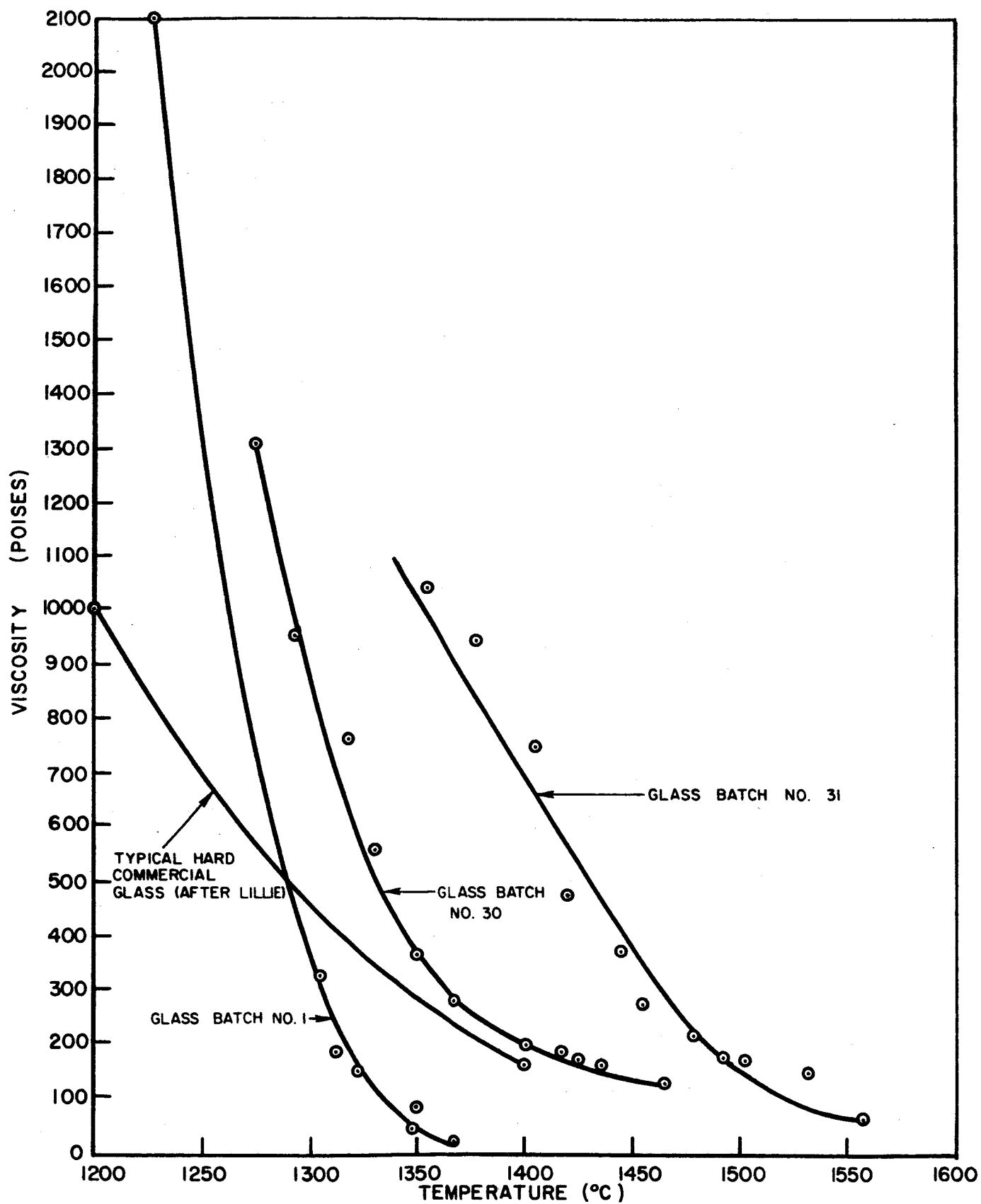
EXTRAPOLATED CALIBRATION CURVE FOR N.B.S. VISCOSITY STANDARD "P"



BROOKFIELD VISCOMETER INSTALLED ON TUNGSTEN FURNACE
FOR HIGH TEMPERATURE VISCOSITY MEASUREMENTS



EXPERIMENTALLY DETERMINED VISCOSITY-TEMPERATURE RELATIONS



APPARATUS FOR DETERMINING SHEAR MODULUS BY MEASUREMENT OF THE VELOCITY OF SOUND

